



STUDY ON THE REVERSIBILITY OF EPDM VULCANIZATION

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Reclaiming of EPDM-based rubber is still unexplored; contrary to reclaiming of NR-based rubber. A few attempts have been made to reverse the vulcanization of EPDM rubber, but the curing activity of the resulting reclaim was low: sulfur crosslinks in the cured material were broken, but without restoration of the active moieties for re-vulcanization. Additionally, the breakdown process was not selective and polymer chains were broken together with crosslinks. A thorough investigation of the influence of temperature, time, shear and concentration of the reclaiming agent, hexadecylamine HDA, on the devulcanization efficiency is done. Out of these four variables, devulcanization temperature clearly has the largest influence. HDA is suitable as devulcanization agent for different types of EPDM compounds, but an optimization of the devulcanization parameters is crucial. A mechanistic study shows that the crosslink distribution of the cured rubber also influences the devulcanization efficiency and determines the characteristics of the reclaim.

Introduction

The extensive utilization of rubber in various applications, e.g. in the automotive sector, causes a problem in terms of rubber waste. Finding a solution for this problem is a challenge for the rubber industry nowadays, especially regarding the upcoming European legislation concerning automotive vehicles recycling¹.

Rubber can be recycled in different ways, of which the most common variant is the reuse of ground cured rubber. However, upgrading the quality of the ground material by devulcanization is of interest as it broadens the possibilities for the utilization of the recycled material. In devulcanization processes, the sulfur network is broken and the cured rubber is converted into a material, which ideally can be processed like the original unvulcanized compound. However, the currently used reclaiming processes result in a combination of crosslink-scission and main-chain scission of the constitutive polymer chains in the rubber networks, caused by heat, shear and chemical reactions.

A clear distinction has to be made between devulcanization and reclaiming:

- Devulcanization: only crosslinks are broken, the hydrocarbon backbone remains intact.
- Reclaiming: crosslink scission as well as main-chain scission occurs.

Rubber-reclaim is used in a wide variety of products ranging from floorcoverings with a high percentage of reclaim, to tires with a low percentage. The reasons for using rubber-reclaim are, besides the cost reduction, processing advantages such as shorter mixing, milling and extrusion cycles. The most important advantage of using reclaim in cured articles is an improvement of the aging resistance^{2,3}.

Reclaiming of sulfur-cured natural rubber (NR) is a common technology for many decades already. Earlier attempts to apply the same technology to the recycling of cured EPDM-rubber – automotive parts like window- and door seals, resp. radiator hoses, and building and construction articles like roof sheetings and window profiles – were not successful, because the chemistry of the NR-devulcanization could not simply be transposed onto EPDM. Contrary to NR-based rubber, reclaiming of EPDM-based rubber is still very unexplored, but it is an interesting topic in view of the continuous market growth of EPDM. Former work at the University of Twente^{4,5} has shown that sulfur-cured EPDM needs far more strenuous conditions to devulcanize than NR. Diaryldisulfides, commonly used as devulcanization-aids for NR, are to some extent effective in promoting the devulcanization of EPDM. It results however in EPDM-devulcanizates, of which the chemical integrity is largely damaged and which are insufficiently active in re-vulcanization. α -H-containing aliphatic amines are promising alternatives as effective devulcanization agents for EPDM-rubber.

In the present paper specific attention will be paid to the devulcanization of EPDM-rubber. Conventionally (CONV) and efficiently (EV) vulcanized EPDM-rubbers are devulcanized with Hexadecylamine (HDA) as devulcanization agent under various conditions. The influence of temperature, time, shear and amount of reclaiming agent on the devulcanization of EPDM-rubber is investigated.

Experimental

An EPDM-rubber masterbatch was premixed on large scale by DSM Elastomers B.V., the Netherlands. This masterbatch had a composition and was vulcanized according to the recipe shown in Table I, a typical recipe for an automotive door seal profile. Devulcanization was performed on these two materials after grinding.

Table I – Composition of masterbatch and curing recipes

Material	Compound A CONV system (phr)	Compound B EV system (phr)
Masterbatch		
EPDM		100
ZnO		5
Stearic acid		1
Carbon black N550		70
Carbon black N772		40
Paraffinc oil		70
Curing additives		
TMTD	1.0	1.0
MBT	0.5	
CBS		1.5
DPTT		0.8
Sulfur	1.5	

Materials

The EPDM used within this study was Keltan 2340A. Sulfur and 2-mercaptobenzothiazole (MBT) (Merck), tetramethylthiuram disulfide (TMTD), N-cyclohexyl-2-benzothiazole sulfenamide (CBS) and dipentamethylene thiuramtetrasulfide (DPTT) (Flexsys, the Netherlands) were used as curatives for the vulcanization. Hexadecylamine, 92% pure (HDA) (Merck) was used as devulcanization agent. Paraffinic oil (Sunpar 2280, Sunoco Inc.) was used as reclaiming oil, the same as used for the original compound. Decahydronaphtalene, a mixture of the cis- and trans-isomer with a purity of 98%, and petroleum ether, 40-60°C (Acros), acetone and tetrahydrofuran (THF) (Biosolve) were used as solvents. 2-Propanethiol, ≥98% pure (Merck), 1-hexanethiol, 95% pure, piperidine, 99% pure and n-heptane, 99% pure (all from Aldrich) were used for the specific crosslink distribution determinations.

Mixing and Vulcanization

The curing additives shown in Table I were mixed into the EPDM-rubber masterbatch on a Schwabenthan two-roll mill, Polymix 80. The cure characteristics were determined with a Rubber Process Analyzer RPA 2000 from Alpha Technologies and the blends were vulcanized in a Wickert laboratory press 1600/5x4/3 at 100 bar for the optimum curing time t_{90} , at 170°C.

Grinding

250x250x6mm vulcanized EPDM-rubber sheets were cryogenically ground in a Bauknecht grinder. The rubber samples were immersed in liquid nitrogen before grinding and then ground to particles with a size ≤ 8 mm.

Design of Experiments

A statistical approach was taken to evaluate the most influential factors for the devulcanization of the EPDM-vulcanizates. Changing the value of every separate factor characterizing the process, one at a time, until no further improvement is accomplished, makes the effort and experimental cost required for such a study prohibitive. The solution is to construct a carefully selected set of experiments, in which all relevant factors are then varied simultaneously.

Table II – Devulcanization conditions

Parameter	Range
Temperature	225-275°C
Time	5-10 minutes
Rotor speed	50-100 rpm
Amount of devulcanization agent	0-10 wt%

As the relative importance of the factors was not known a priori, a Design of Experiments (DOE) was adopted⁶. Prior to doing any experiments, the input conditions such as the number of variable factors and their ranges (see Table II), the number of responses (Mooney viscosity, crosslink density and insoluble fraction) and the experimental objective should be specified. Then the experimental design is elaborated and the experiments are carried out. Each experiment gives values of the responses as result. Thereafter, these data are analyzed by regression analysis.

This results in a model equation relating the changes in the responses to the changes in the factors. The model indicates which factors are important, and how they combine in influencing the responses⁶. To optimize the parameter settings for the most complete devulcanization, Response Surface Modeling was used. The objective of the optimization is:

1. To predict the response values for all possible combinations of factors within the experimental region, and
2. To identify an optimal experimental point.

MODDE 6.0, the software from Umetrics, was used to model the response properties. For this investigation, a central composite face-centered (CCF) design, a quadratic model, is the appropriate one and the chosen parameters and variables result in the experimental design as shown in Table III. In cases when the experimental results were too skewed to result in a workable model, it was necessary to perform a logarithmic transformation of the responses.

Table III – Experimental design for EPDM-rubber devulcanization

Exp. No.	Temperature (°C)	Time (min)	Rotor speed (rpm)	Amount of HDA (wt%)
1	225	5	50	0
2	275	5	50	0
3	225	5	100	0
4	275	5	100	0
5	225	10	50	0
6	275	10	50	0
7	225	10	100	0
8	275	10	100	0
9	225	5	50	10
10	275	5	50	10
11	225	5	100	10
12	275	5	100	10
13	225	10	50	10
14	275	10	50	10
15	225	10	100	10
16	275	10	100	10
17	225	7.5	75	5
18	275	7.5	75	5
19	250	7.5	50	5
20	250	7.5	100	5
21	250	5	75	5
22	250	10	75	5
23	250	7.5	75	0
24	250	7.5	75	10
25	250	7.5	75	5
26	250	7.5	75	5
27	250	7.5	75	5

Devulcanization

The cured EPDM-rubber compounds were devulcanized in a Brabender Plasticorder PL 2000. Its mixing chamber had a volume of 35 cc and a cam-type rotor. The fill factor was 0.7. The devulcanization agent used was HDA and the parameters temperature, time, rotor speed and amount of devulcanization agent were varied as shown in Table II. 5 wt% of reclaiming oil relative to the vulcanizate was used for each experiment. The reclaims based on compounds A and B are further referenced as reclaim A and reclaim B, respectively. The molecular structure of the devulcanization agent is shown below:



Characterization of Devulcanizates

Mooney Viscosity – The Mooney viscosity ML(1+4)125°C was determined using a Mooney viscometer MV2000 VS from Alpha Technologies according to ISO R289.

Extractions– The reclaimed samples were extracted in a Soxhlet apparatus, first for 24 hrs with acetone to extract the polar substances, and then for 48 hrs with THF to remove debound/devulcanized polymer. The completion of the extraction was checked by drying the samples in a vacuum oven and determining the weight loss until no further significant amount of solubles could be extracted. The amount of left-over is defined as the insoluble fraction.

Crosslink Density Determinations – Equilibrium swelling measurements were performed on extracted samples: the crosslink density of the insoluble fraction only was determined. The elastically active network chain density of the insoluble fraction was measured in decahydronaphtalene for 72 hrs at room temperature. The weight of the swollen vulcanizates was determined after removal of surface liquid with absorption paper. The overall crosslink density was calculated according to the Flory-Rehner equation⁷. The Kraus correction⁸ for carbon black was not applied, because the

results were compared in a relative manner in the same recipe. The Huggins interaction parameter for the EPDM/decahydronaphtalene interaction was determined by Dikland⁹.

The specific crosslink distribution in the vulcanizates and the insoluble fraction of the reclaims were studied by using thiol-amine reagents. A solution of 0.4M 2-propanethiol and 0.4M piperidine in n-heptane and a reaction time of two hours were used for breaking polysulfidic crosslinks; poly- and disulfidic crosslinks were broken in a 1M 1-hexanethiol/piperidine solution within 48 hrs. For a detailed description of this method see Campbell and Saville and others¹⁰⁻¹³.

Light Microscopy – 0.5 grams of reclaim A and B, devulcanized at 250°C at a rotor speed of 75 rpm for 7.5 min with the addition of 5 wt% of HDA, was mixed into virgin EPDM-polymer on a two roll mill. The material was put under a light microscope under magnifications of 5-20x.

Results

The crosslink density, specific crosslink distribution and insoluble fraction of the compounds A and B after vulcanization and before devulcanization are given in Table IV.

Table IV – Crosslink density and distribution of the original compounds

Feed stock	Crosslink density $\times 10^{-4}$ mol/cm ³ (%)			Insoluble fraction (%)
	Overall	Monosulfidic	Di- and polysulfidic	
Compound A	5.32 (100%)	0.81 (15%)	4.50 (85%)	75.1
Compound B	2.23 (100%)	1.17 (52%)	1.06 (48%)	71.9

Compound A is primarily characterized by poly- and disulfidic crosslinks and contains only 15% monosulfidic bonds. Compound B on basis of the EV curing system has a similar share of poly- together with disulfidic crosslinks and monosulfidic bonds. The insoluble fraction of compound A is slightly higher than that of compound B.

To obtain a good representative model in this case, it was necessary to perform a logarithmic transformation on the monosulfidic crosslink density results of reclaims A and B, and on the Mooney viscosity of reclaim A. The other responses did not need a transformation.

Table V – Quality of the specific mathematical models

Model	Q ²	R ²	R ² _{adj.}
Reclaim A			
Mooney viscosity	0.937	0.982	0.970
Insoluble fraction	0.938	0.968	0.954
Overall crosslink density	0.683	0.802	0.760
Monosulfidic crosslink density	0.723	0.858	0.810
Reclaim B			
Mooney viscosity	0.941	0.971	0.961
Insoluble fraction	0.889	0.952	0.936
Overall crosslink density	0.785	0.916	0.883
Monosulfidic crosslink density	0.813	0.890	0.864

Table V shows the quality of the specific models. Q² values higher than 0,5 imply a good model and Q² values above 0,9 imply an excellent model. The models for Mooney viscosity and insoluble fraction after devulcanization of both compounds fit very well; the models for the overall and monosulfidic crosslink densities of both systems are good as well, but show a slightly larger deviation. To check the validity of the model, extra experiments were executed with factor settings different from those given in Table III. These experimental results coincided with the predicted results on basis of the mathematical models within 95% probability. It can be concluded that the mathematical models are able to predict the results of experiments within the chosen experimental window with good accuracy.

Reclaim A

The influence of all variable factors on the Mooney viscosity, insoluble fraction and crosslink density of reclaim A is depicted in Figures I-III. Constant parameter settings are rotor speed: 75 rpm, devulcanization time: 7.5 minutes, temperature: 250°C and HDA concentration: 5 wt%.

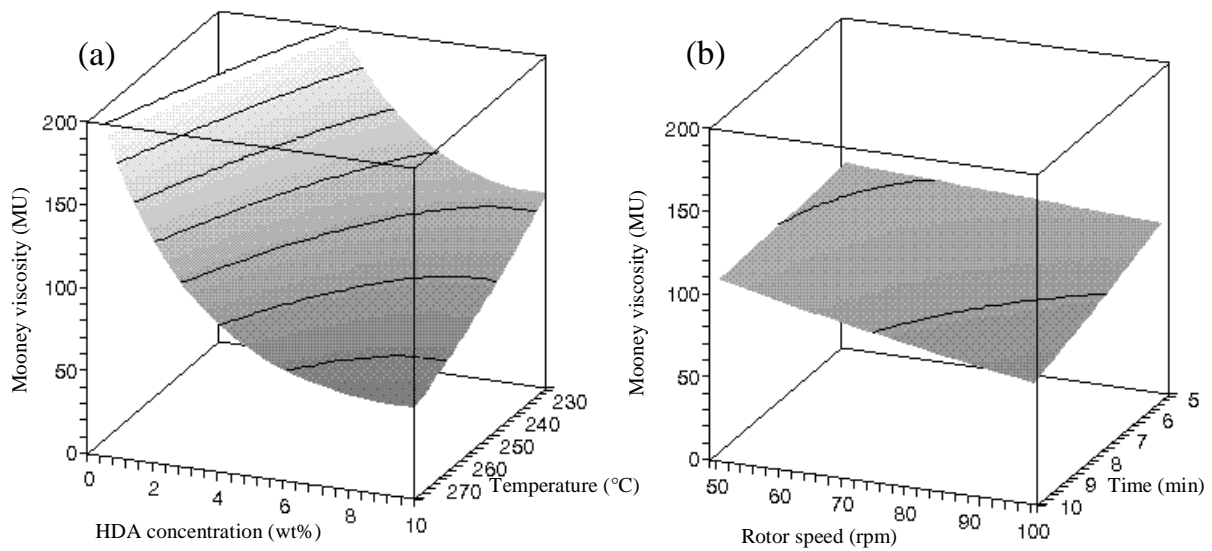


Figure I – Influence of (a): HDA concentration and temperature and (b): rotor speed and time on the Mooney viscosity of reclaim A.

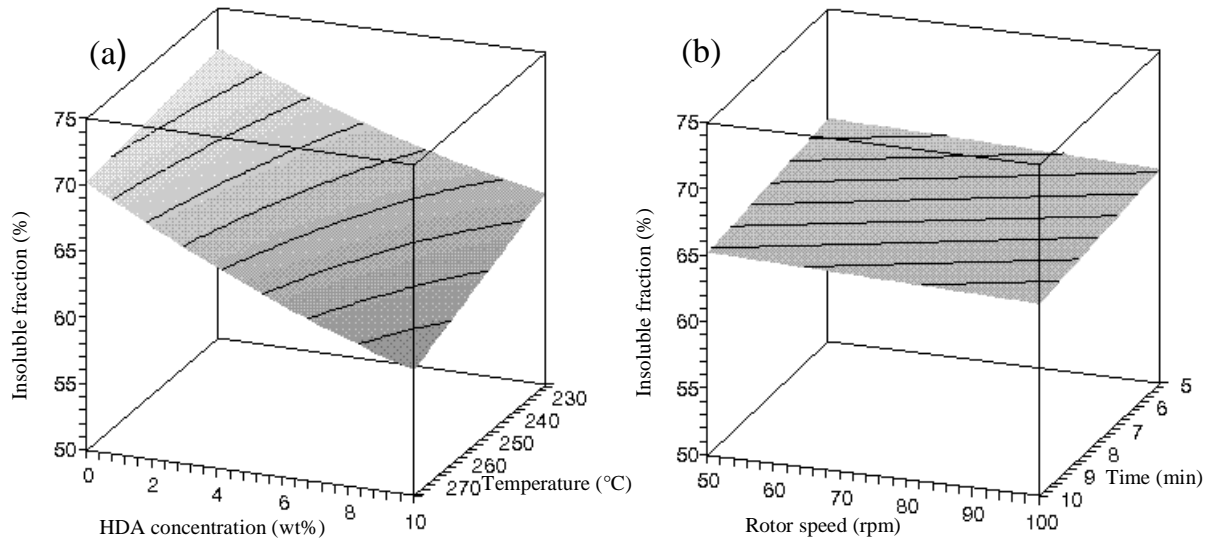


Figure II – Influence of (a): HDA concentration and temperature and (b): rotor speed and time on the insoluble fraction of reclaim A.

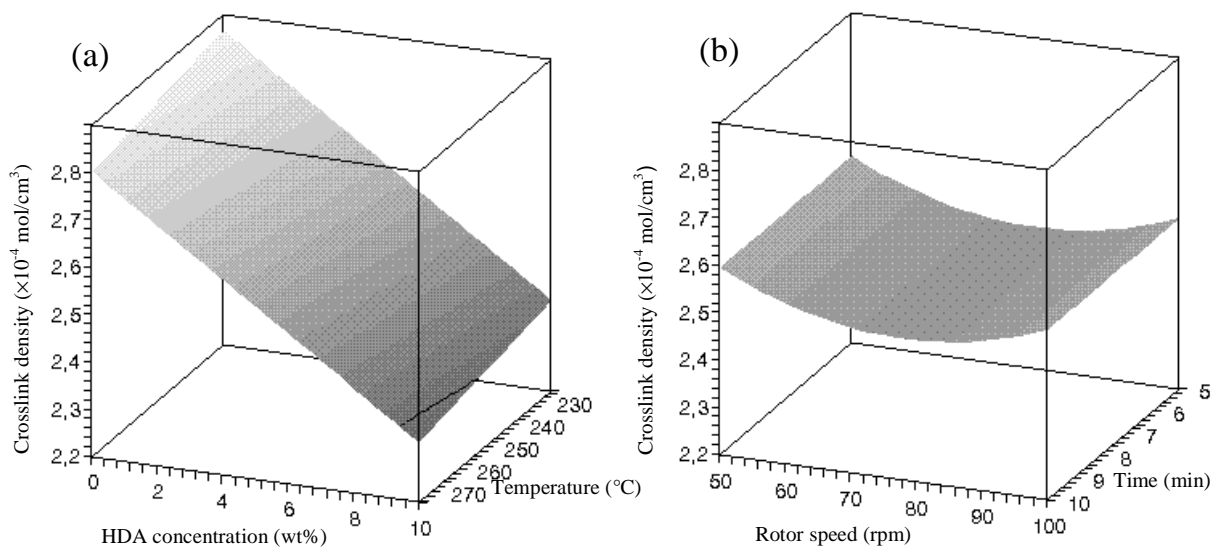


Figure III – Influence of (a): HDA concentration and temperature and (b): rotor speed and time on the crosslink density of reclaim A.

Figures I-III show that the influence of HDA concentration and temperature on the Mooney viscosity, insoluble fraction and crosslink density is quite significant, while the influence of rotor speed and devulcanization time on these responses is very little. For this reason further focus will be on HDA concentration and temperature.

Figure IV shows in more detail the influence of HDA concentration and temperature on the Mooney viscosity, insoluble fraction and crosslink density of reclaim A.

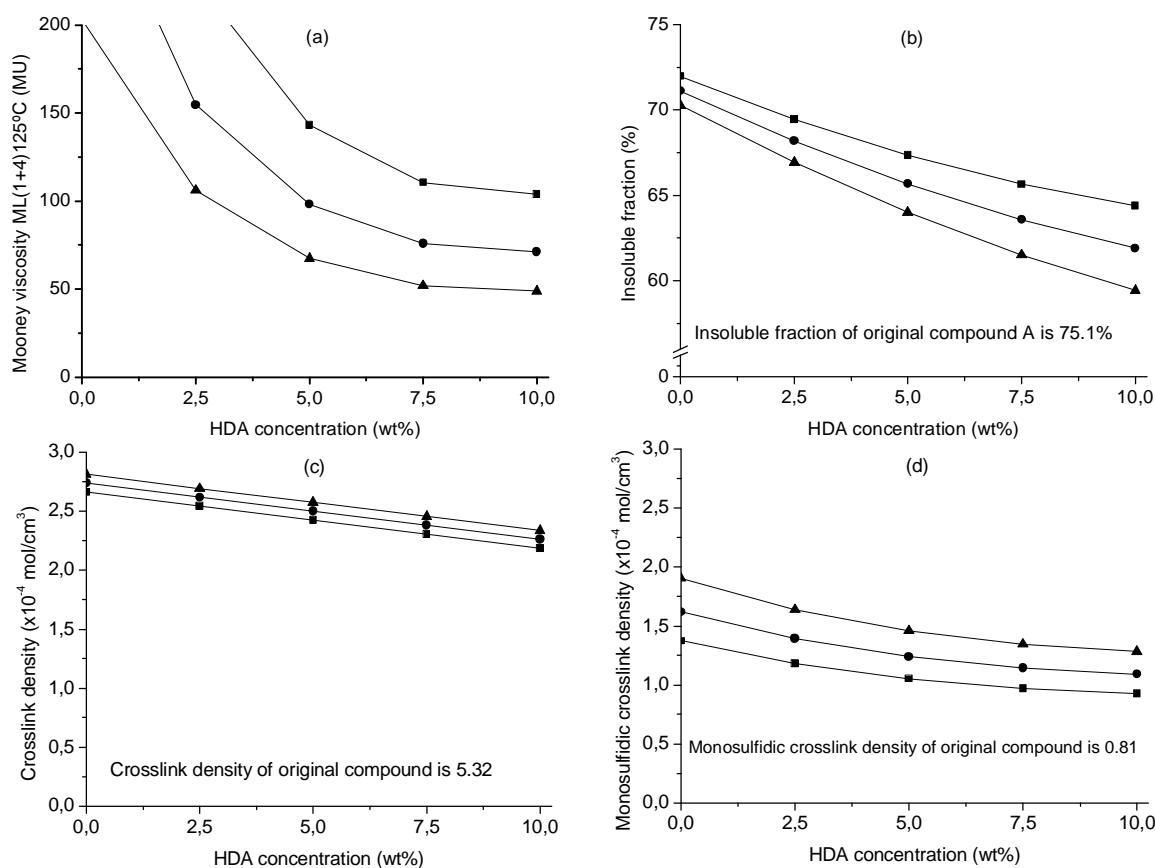


Figure IV – Reclaim A: Influence of HDA concentration and temperature on (a): Mooney viscosity; (b): insoluble fraction; (c): overall crosslink density and (d): monosulfidic crosslink density of reclaim A. (■): 225°C; (●): 250°C and (▲): 275°C. Devulcanization time is 7.5 minutes at a rotor speed of 75 rpm.

Figure IV(a) shows that for reclaim A, the influence of the HDA concentration on the Mooney viscosity is very significant up to a concentration of approximately 5wt% HDA, but levels off at higher concentrations at all temperatures. Without the addition of HDA, the predicted Mooney values are above 200 MU, which is out of the measuring range of the Mooney viscometer. In Figure IV(b), the insoluble fraction of reclaim A decreases with increasing concentration of HDA and increasing temperature. At higher concentrations of HDA, the difference in insoluble fraction between the different temperatures is more significant than at lower concentrations HDA. Figure IV(c) shows the crosslink density versus the concentration HDA at different temperatures: with increasing HDA concentration a linear decrease in crosslink density is obtained for reclaim A. With increasing temperature the initial reduction of the crosslink density is reduced. Without the addition of HDA the crosslink density of reclaim A already decreases from the original value of 5.32 to approximately $2.7\text{--}2.8 \times 10^{-4}$ mol/cm³ for all temperatures. The main decrease in crosslink density for reclaim A is therefore caused by the mere thermal process. Addition of HDA only helps to devulcanize the compound a little bit more. Figure IV(d) shows that the monosulfidic crosslink density decreases with increasing concentration of HDA at all temperatures. The amount of monosulfidic crosslinks after reclaiming is in all cases significantly higher than the value of the original compound A.

Figure V shows light microscopic pictures of reclaim A: the reclaim particles with a diameter larger than 1 mm are visible. Figure V(b) shows the sharp edges of one particle in a matrix of virgin EPDM.

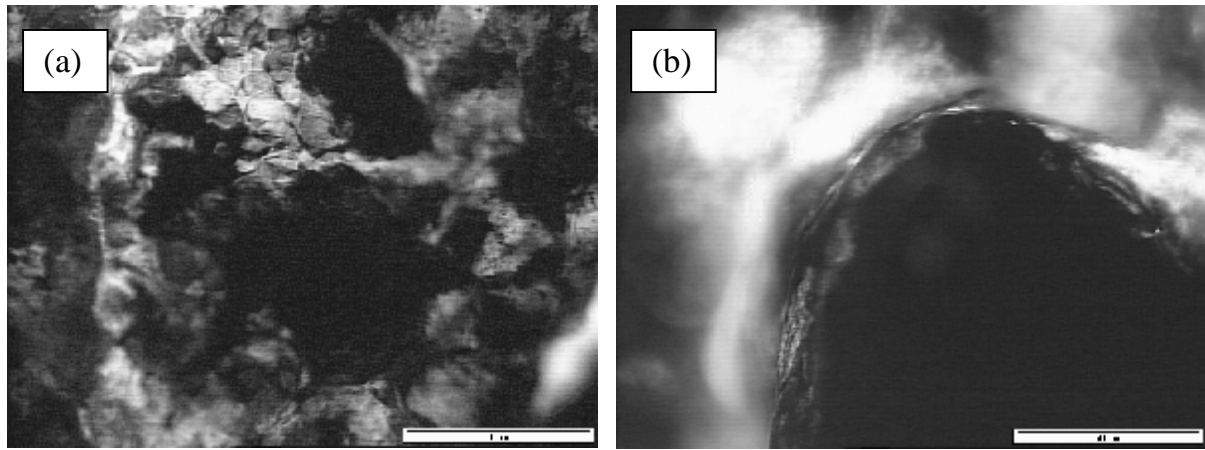


Figure V – Microscopic picture of reclaim A with a (a): 5x magnification and (b): 10x magnification.

Reclaim B

For compound B, additional experiments were executed at 200°C. At 275°C the rubber material was extensively broken down resulting in a low viscous and tacky behavior. Therefore extra experiments were performed at the lower temperature range in order to obtain a higher viscous and less tacky rubber reclaim.

For reclaim B the same trends are valid as seen earlier for reclaim A: a significant influence of HDA concentration and temperature and very little influence of rotor speed and devulcanization on the responses. Again, further focus will be on HDA concentration and temperature only.

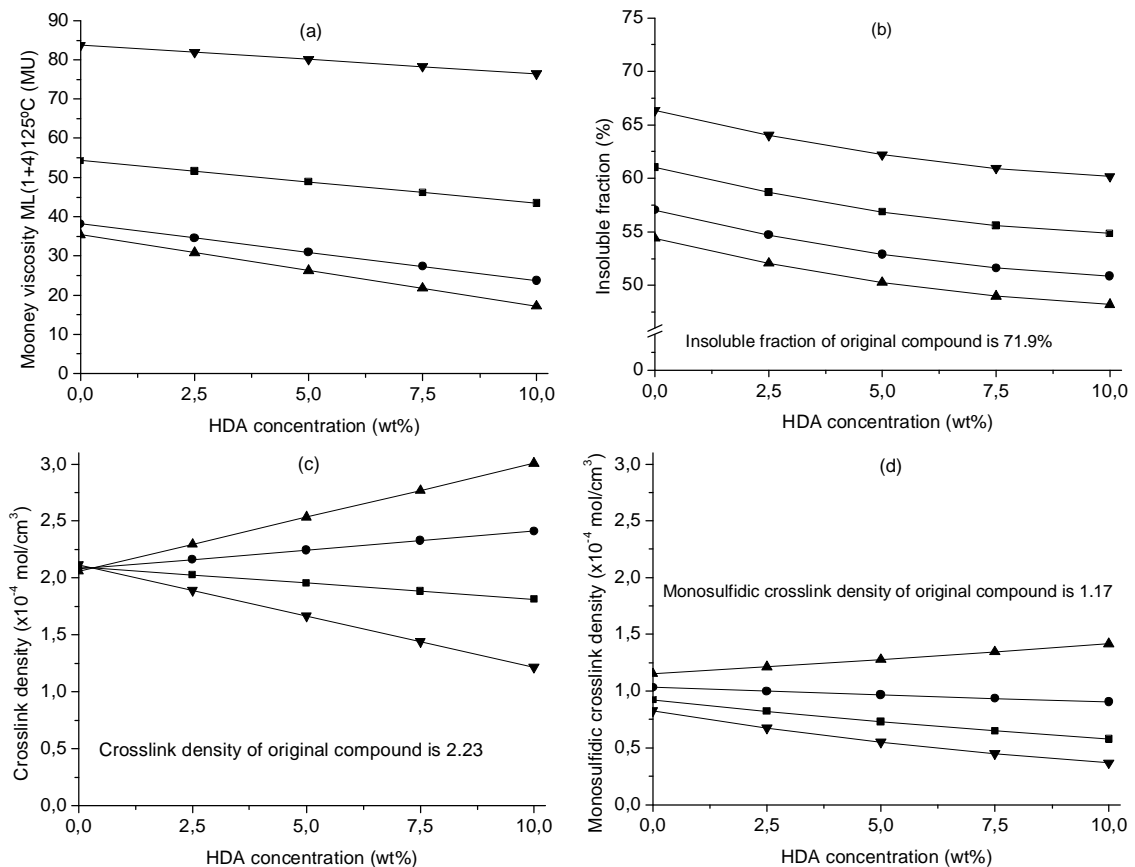


Figure VI – Reclaim B: Influence of HDA concentration and temperature on (a): Mooney viscosity; (b): insoluble fraction; (c): overall crosslink density and (d): monosulfidic crosslink density of reclaim B. (▼): 200°C; (■): 225°C; (●): 250°C and (▲): 275°C. Devulcanization time is 7.5 minutes at a rotor speed of 75 rpm.

Figure VI(a) shows a linear decrease in Mooney viscosity with increasing HDA concentration for reclaim B. The influence of temperature on the Mooney viscosity levels off with increasing temperature. From Figure VI(b) can be seen that the insoluble fraction decreases with increasing temperature and increasing HDA concentration. Figure VI(c) shows, contrary to reclaim A, that the crosslink density of the insoluble fraction without addition of HDA is still equal

to the crosslink density of the original vulcanized compound B for all temperatures: thermal treatment alone does not lead to a decrease of the crosslink density in this case. The combination of a moderate reclaiming temperature and the addition of HDA as reclaiming agent is required for a significant decrease in crosslink density: at 200°C and 225°C, a decrease in crosslink density with increasing concentration HDA is observed. At reclaiming temperatures of 250°C and 275°C, an increase in crosslink density is observed in spite of addition of HDA. After devulcanization at 275°C reclaim B has a lower viscosity compared to reclaim A and shows a tacky behavior, while the crosslink density of the insoluble fraction increased with increasing concentration HDA. Figure VI(d) shows the influence of the HDA concentration and temperature on the monosulfidic crosslink density of reclaim B. During devulcanization of compound B at 275°C, an increase in monosulfidic crosslink density with increasing HDA concentration is observed, while at 200°C, 225°C and 250°C the amount of monosulfidic crosslinks decreases with increasing HDA concentration. The actual concentration of monosulfidic crosslinks at the three lowest temperatures is even lower than the monosulfidic crosslink density of the original compound B.

In Figure VII(a), particles of reclaim B with a diameter of less than 500µm can be seen, and Figure VII(b) shows that these reclaim particles do not have the sharp contours as seen for reclaim A in Figure V: the particles of reclaim B are more likely to be smeared out when mixed into a rubber compound.

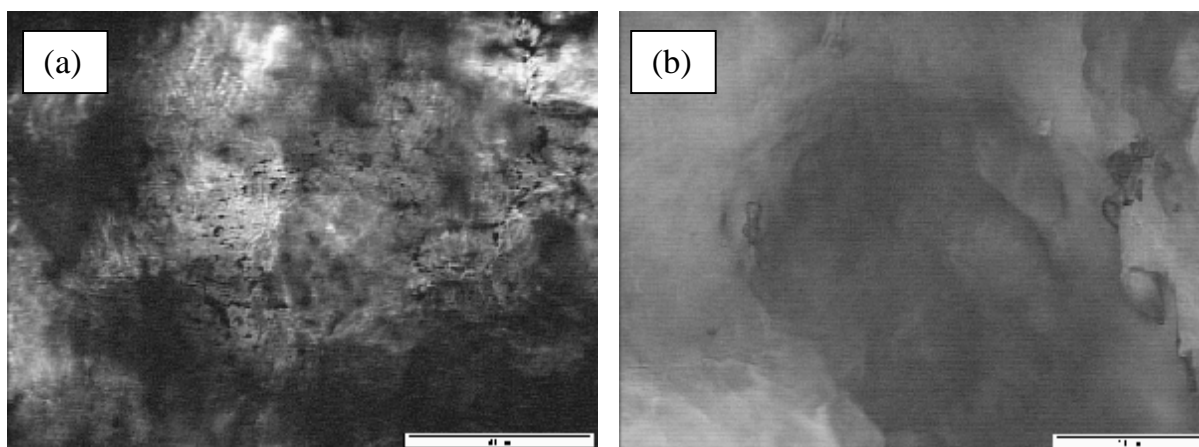


Figure VII – Microscope picture of reclaim B with a (a): 10x magnification and (b): 20x magnification.

Discussion

It is well known that monosulfidic crosslinks have a higher dissociation energy than di- or polysulfidic bonds. Consequently it is expected that vulcanizates with a higher percentage of poly- and/or disulfidic crosslinks will be easier to be devulcanized than those with many monosulfidic bonds. This would favor compound A for easier devulcanization than compound B. However, the outcome of this study is not in accordance with this expectation.

A conspicuous difference is observed in the reclaiming/devulcanization of compounds A and B in Figures IV and VI. For compound B even without use of HDA, by simple thermal treatment already a significant decrease of the viscosity into the measurable range is obtained. For compound A thermal treatment is insufficient and addition of HDA is required. One possible explanation for this difference is the appr. 2.5 times higher overall crosslink density of compound A before reclaiming, as also represented in a slightly larger insoluble fraction. Another difference is that the viscosity decrease levels off for higher HDA concentrations, represented by di- resp. converging curves for compound A as seen in Figure IV(a). For compound B a linear decrease in viscosity is found and the curves are essentially parallel (Figure VI). The effect of HDA is apparently entirely different for both compounds.

For compound A thermal treatment with increasing amounts of HDA results in only a relatively small decrease of the insoluble fraction, slightly dependent on the temperature applied. Or conversely, only 3-5% extra generated soluble devulcanized matter is generated compared to the original vulcanized compound A, which eventually results in a measurable Mooney viscosity. However, Figures IV(b) and (c) show, that throughout the insoluble fraction, a significant decrease in crosslink density is reached relative to the initial value. The additional shift from mainly poly- and disulfidic crosslinks to some more monosulfidic crosslinks, as shown in Figure IV(d) as well, demonstrates that real devulcanization is taking place as opposed to main-chain scission, which otherwise would have given larger amounts of soluble material, without a significant shift in crosslink density and distribution in the insoluble fraction¹⁴.

For compound B basically the opposite effect is seen: significant amounts of soluble material are generated. Dependent on the temperature applied, 6-18% more sol fraction is generated compared to the original vulcanized compound B, while the crosslink density within the non-soluble matter for thermal recycling is essentially still the same as that of the vulcanized material. This is a typical behavior for a vulcanizate which becomes plasticized during reclaiming by main-chain scission. Dependent on the reclaiming temperature, the crosslink density de- or increases after addition of HDA as devulcanization agent.

Both vulcanizates, the conventional polysulfidic one and the efficiently vulcanized primarily monosulfidic one, can be reclaimed. Temperature is the main governing factor and addition of HDA significantly enhances the effects. Rotor speed as well as treatment time have a relatively small effect. Saturation in reclaiming is reached quite fast with hardly any further generation of sol fraction after a few minutes. The different behavior between the two compounds is schematically depicted in Figure VIII.

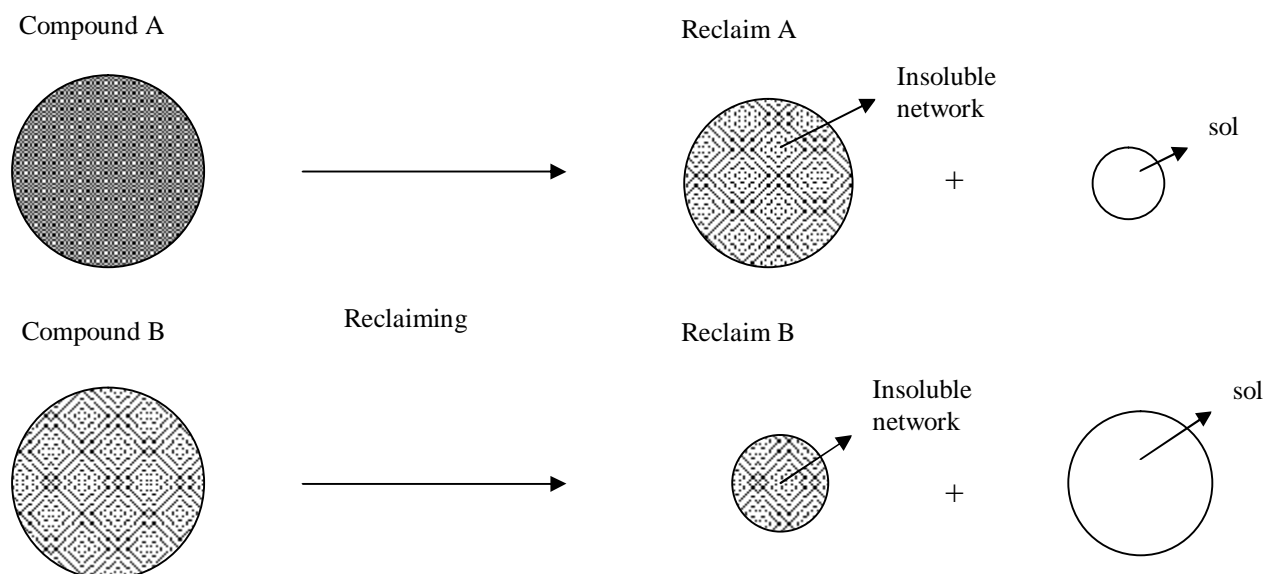


Figure VIII – Schematic sketch of the different reclaiming behavior of compound A and B.

For compound A, mainly devulcanization, a breakdown of the crosslink network, takes place and leads to additional soluble material. The particles subjected to reclamation remain grossly intact, as shown in Figure V. For compound B, main-chain scission peels off low molecular weight material from the outside of the particles, these particles shrink in size, while the crosslink density within the particles remains basically unchanged: see Figure VII, wherein small particles with unclear contours are seen.

The difference between compound A and B during the devulcanization process can only originate from the different concentrations of monosulfidic crosslinks versus di- and polysulfidic ones. Under the reclaiming conditions used in these experiments, sufficient heat is transferred to break the low energetic poly- and disulfidic bonds. The bond strength of monosulfidic crosslinks approaches that of saturated carbon-carbon bonds, and as there is an enormous surplus of carbon-carbon bonds relative to the amount of sulfur crosslinks, the chance of breaking small oligomeric fragments out of the total network by main-chain scission is dominant. The fact that some monosulfidic crosslinks are formed during the reclaiming of compound A is here of minor importance. A more in-depth investigation of this argumentation will be the subject of a following paper.

In case of the efficiently vulcanized compound B, a proper combination of temperature and HDA as devulcanization agent is of primary importance in order to prevent an increase in crosslink density in the insoluble fraction. As seen in Figure VI(c): at lower temperatures like 200°C and 225°C the crosslink density in the insoluble fraction is decreased with addition of HDA, while at higher temperatures it is even increased. Former work has demonstrated that HDA can also act as a crosslinking promoter for EPDM¹⁵⁻¹⁷, dependent on the crosslink distribution. Amines in general have an enhancing effect on sulfur crosslinking. Therefore, a proper combination of a relatively low temperature and the addition of HDA as devulcanization agent is crucial in order to obtain a high-quality reclaim from this compound. Figures IV(d) and VI(d) clearly indicate that HDA is able to break even monosulfidic crosslinks to a certain extent and to lower the crosslink density of reclaim A and B.

Conclusion

The efficiency of devulcanization of sulfur vulcanized EPDM compounds with hexadecylamine strongly depends on the crosslink density and crosslink distribution of the original EPDM compounds.

During devulcanization of a conventionally cured, thus primarily poly- and disulfidic EPDM-vulcanizate, a significant reduction in crosslink density down to 50% of the original value is obtained within the temperature range of the experiments: 225°C till 275°C. Thermal devulcanization hardly has any effect: it results in only a slight increase in soluble fraction and the material still has a Mooney viscosity above the measuring limit. Within the whole temperature range, the addition of hexadecylamine significantly helps to decrease the crosslink density. Primarily poly- and disulfidic crosslinks are broken, and some crosslinks are transformed into monosulfidic bonds.

The devulcanization process of an efficiently vulcanized material with similar percentages of monosulfidic and longer crosslinks shows different characteristics. Heat together with HDA as devulcanization agent causes random main-chain scission of the rubber network, resulting in small oligomeric molecules released from the network. The crosslink density of the non-soluble material responds to the addition of HDA with a decrease in crosslink density at low temperatures like 200°C and 225°C, but in an increase at higher temperatures. HDA is even able to break monosulfidic crosslinks. Reclaiming of the conventional compound therefore takes place primarily by sulfur crosslink scission or devulcanization, while reclaiming of the efficiently vulcanized compound occurs mainly by main-chain scission. Crosslink scission will also take place but is of minor importance relative to the abundant scission of carbon-carbon bonds.

Only little influence of rotor speed and devulcanization time on the reclaiming of both types of compounds is noticed within the experimental frame, as represented by the basically constant Mooney viscosities and insoluble fractions of the reclaims.

HDA is a valuable devulcanization aid for different EPDM compounds, taken into account the different devulcanization parameter settings for each single compound. Of the four variable factors examined, devulcanization temperature clearly has the largest influence on the devulcanization within the experimental ranges investigated.

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